

## A NEW METHOD OF ELECTROPOLISHING NIOBIUM \*

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By a new method of electropolishing niobium we have obtained very smooth surfaces. In electropolished TE<sub>011</sub>-cavities with an anodic oxide film a  $Q$ -value of  $3 \times 10^{10}$  and a critical magnetic field of 80 mT were obtained in the X-band without any heat-treatment.

There are two ways of producing microscopically smooth and damage-free finishes on niobium, namely by chemical and electrolytic polishing. Mechanical methods can produce smooth finishes, but only with a high concentration of lattice defects and impurities. Where shapes are complicated, chemical polishing has its limitations since the specimens have to be immersed in the solution under defined conditions of solution flow etc. Local disturbance of the solution flow results in etching instead of polishing at such points. In such a case, electropolishing is to be preferred. The potential distribution between the anode and the cathode can generally be adapted to the geometry of the specimen (anode).

A large number of electropolishing solutions are known [1,2], which would point to the fact that a special method is necessary for a specific geometry or a specific physical state of the niobium. However, the methods employed so far have the disadvantage that etching is observed when removing layer thickness of, for instance, 100  $\mu\text{m}$ . In many cases, however, it is necessary, e.g. for the complete removal of damage layers resulting from machining, to remove layers of up to 0.5 mm thick and polish the surface at the same time.

The method under discussion offers this possibility, together with a number of other advantages. The electrolyte used consists of 95 to 97% sulphuric acid and 40% hydrofluoric acid in a ratio of 85 : 10 by volume. The best bath voltage was between 9 and 15 V, and depends on the solution concentration, the bath temperature and the electrode geometry. Unlike many polishing techniques, the optimum polishing conditions are not governed by the plateau voltage in the current-voltage characteristic, but are characterised by a current oscillation with decreasing amplitude

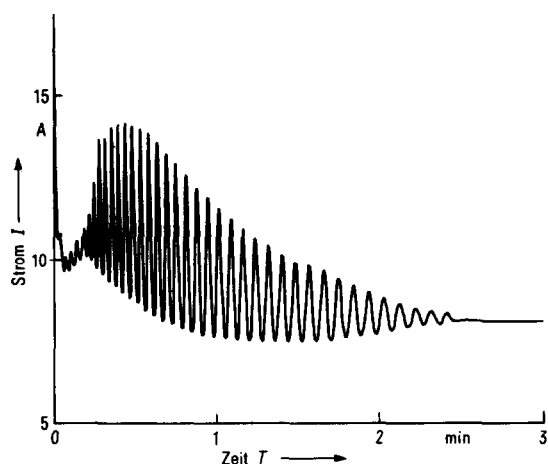


Fig. 1. Electropolishing niobium current oscillations.

in the above-mentioned voltage range. Fig. 1 shows the typical characteristic of this oscillation. The voltage associated with the current oscillations must be controlled at a constant value. The mean current density is of the order of 100 mA/cm<sup>2</sup>.

The oscillations reflect the phenomena at the anode, i.e. the building up and partial decrement of an oxide film. The mean thickness of this oxide film tends to increase and results in a current decreasing. The voltage is switched off after the oscillations have decayed and switched on again after an interval of at least 4 minutes during which the oxide film is dissolved by the hydrofluoric acid component of the electrolyte. The oscillations then recommence. This procedure

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can be repeated as often as desired. The bath temperature is chosen between 25 and 35°C and controlled at a constant value. Pure aluminium was used for the cathode and for the anode holder.

The method has the following advantages:

1. By frequently repeating the oscillation operation according to fig. 1, layers of any desired thickness can be removed without the surface finish initially achieved being lost or diminishing.
2. Surface roughness can be removed down to a residual value of approximately  $30\text{ }\mu\text{m}$ . This also applies for the immediate environment of grain boundaries.
3. The cathode geometry need not be matched to the anode geometry since the voltage drop in the anode layer is about 90%. The method therefore polishes true to profile down to surface irregularities of about 0.2 to 0.3 mm.
4. Any parameter changes during the polishing operation, e.g. changes in the concentration of the solution components, manifest themselves in a deviation from the oscillation configuration shown in fig. 1, and can generally be compensated by varying the other parameters (temperature, voltage) provided the deviation is not too great. The compensation procedure can be checked by comparison with the actual oscillations shown in fig. 1.
5. The polishing process can be fully automated.

The method was used for the surface preparation of superconducting niobium cavities. Following precision machining, a pot shaped  $\text{TE}_{011}$ -cavity with a flange for the upper end-plate was electropolished, the layer removed being  $200\text{ }\mu\text{m}$  thick. Subsequent to electropolishing, the  $Q$  value and critical magnetic field were  $Q_0 = 3 \times 10^9$  and  $H_c^{\text{ac}} = 46\text{ mT}$ , respectively, at 1.3 K. After that this electropolished cavity was oxidized by a method published elsewhere [3]. The measurements then yielded the following results:  $Q_0 = 3 \times 10^{10}$ ,  $H_c^{\text{ac}} = 80\text{ mT}$  at 1.3 K. ( $Q_0$ -values always measured just below  $H_c^{\text{ac}}$ .) It should be mentioned that these results were obtained without subjecting the cavity to any degassing or heat treatment. We assume, that the reason for the high values of  $Q_0$  and the critical magnetic field is explained by the fact that a) a very smooth finish is produced by the special electropolishing process, b) the absorbing surface is displaced to a deeper and purer layer by the presence of the oxide film and c) the oxide film suppresses emission effects by dielectric shielding.

#### References

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